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(54) METHOD OF AND APPARATUS FOR ENRICHING SPECIMENS FOR TRACE ANALYSIS BY X-RAY FLUORESCENCE

(71) We, GESELLSCHAFT FUR KERNENERGIEVERWERTUNG IN SCHIFFBAU UND SCHIFFAHRT MBH, a Body Corporate organised under the laws of the Federal Republic of Germany of Reaktorstrasse 1, D-2054 Geesthacht-Tesperhude, Federal Republic of Germany, do hereby declare the invention for which we pray that a patent may be granted to us and the method by which it is to be performed, to be particularly described in and by the following Statement:—

This invention relates to a method of enriching specimens for subsequent trace analysis by X-ray fluorescence with a totally reflecting specimen carrier having a plane surface.

With the known methods of enrichment, that is to say increasing the concentration in the specimen of the trace substance under investigation, considerable difficulties were involved in reducing the detection limit during the trace analysis. These difficulties lay, above all, in the fact that special vessels were used, the contents of which had to be transferred to the specimen carrier after a chemical reaction had been effected. Only relatively large specimen amounts could be treated in these special vessels, and these amounts were often not available, nor were they need during subsequent analysis. Furthermore, losses of trace elements resulted from the influence of the surface of the reaction vessel on the specimen and contamination also occurred during the transfer of the enriched specimen from the reaction vessel to the specimen carrier.

It is an object of the present invention to provide an improved method of enriching specimens which solves the problem of applying the enriched specimen to the specimen carrier.

Accordingly, the present invention consists in a method of enriching specimens for subsequent trace analysis by X-ray fluorescence with a totally reflecting specimen carrier having a plane surface, characterised in that a specimen for enriching is applied directly to the specimen carrier spaced from the margin thereof, appropriate techniques of enriching the specimen being then employed.

The invention is explained in detail below by way of example with reference to the accompanying drawing in which:—

Figure 1 shows a perspective illustration of a specimen carrier with material to be analyzed, and

Figure 2 shows a reaction chamber for the direct treatment of specimens on specimen carriers for X-ray fluorescence analysis.

The specimen carrier of an associated X-ray fluorescence device (as shown for example in DT-OS 26 32 001) illustrated in Figure 1, consists of a plate 1 having a plane-ground surface of about 10 cm^2 of quartz or another suitable material. The specimen material 2, which is not yet enriched, is applied to the surface of the plate 1 so that at most an area of 1 cm^2 in the centre is covered.

An oxidation of the specimen material 2 is carried out on this specimen carrier 1. The specimen on the specimen carrier is exposed, as required, to high or low pressure and temperatures, suitable electromagnetic fields and a suitable gaseous enviroment. The specimen on the specimen carrier may also be treated with small amounts of suitable reagents. If the specimen carrier is first cooled, the amount of specimen which can be applied may be increased if necessary. The specimens on the specimen carriers may also be freeze-dried.

As Figure 2 shows, no difficulties are involved in treating the specimen carrier with material to be analyzed. The specimen carriers, possibly in a plurality, are deposited on the working plate 3 of a substantially gas-tight reaction chamber 4 which may be

provided with a heating or cooling connection 5, gas supply 6, gas outlet 7, vacuum connection 8 and electrodes 9 for an electrical field, for the reactions to be carried out.

The method according to the invention avoids the abovementioned disadvantages and renders it possible to increase considerably the sensitivity which can be achieved with an X-ray fluorescence analysis device. By the method according to the invention, it is possible to work with the smallest amounts of specimen of a few milligrams, which are sufficient for the said analysis devices. The very small amounts of specimen which can be used also lead to a considerable shortening of the reaction times. A further advantage is to be seen in the fact that any chemical reactions which may have taken place incompletely or any insoluble components have substantially no adverse effect on the method because the reaction products remain *in situ* in any case and do not have to be picked up again.

WHAT WE CLAIM IS:—

1. A method of enriching specimens for subsequent trace analysis by X-ray fluorescence with a totally reflecting specimen carrier having a plane surface, characterised in that a specimen for enriching is applied directly to the specimen carrier spaced from the margin thereof, appropriate techniques of enriching the specimen being then employed.

2. A method of enriching specimens for subsequent trace analysis by X-ray fluorescence with a totally reflecting specimen carrier having a plane surface, substantially as herein before described with reference to the accompanying drawings.

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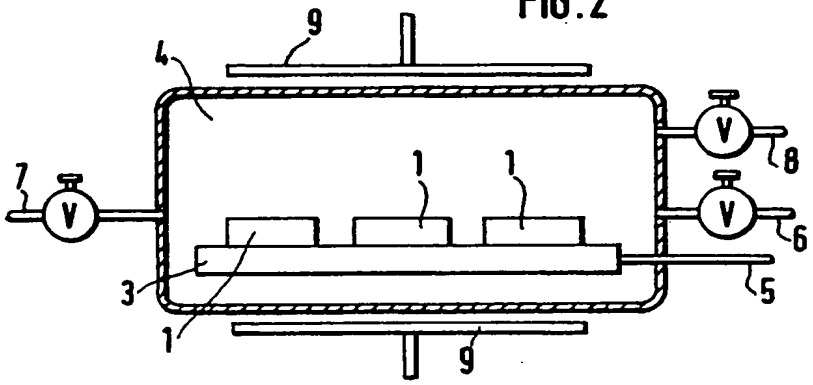
1 SHEET

COMPLETE SPECIFICATION

This drawing is a reproduction of the Original on a reduced scale.

FIG.2



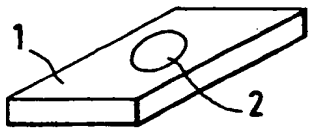


FIG.1

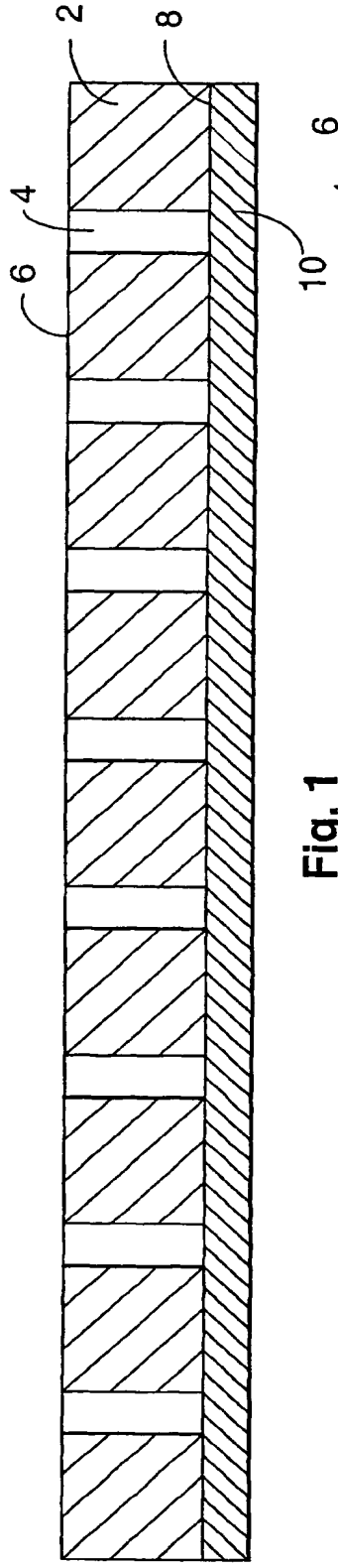


Fig. 1

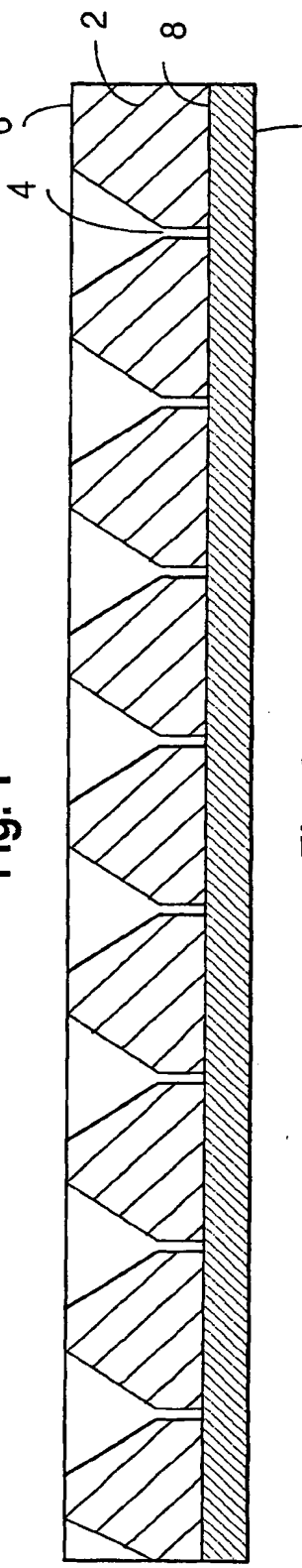


Fig. 2

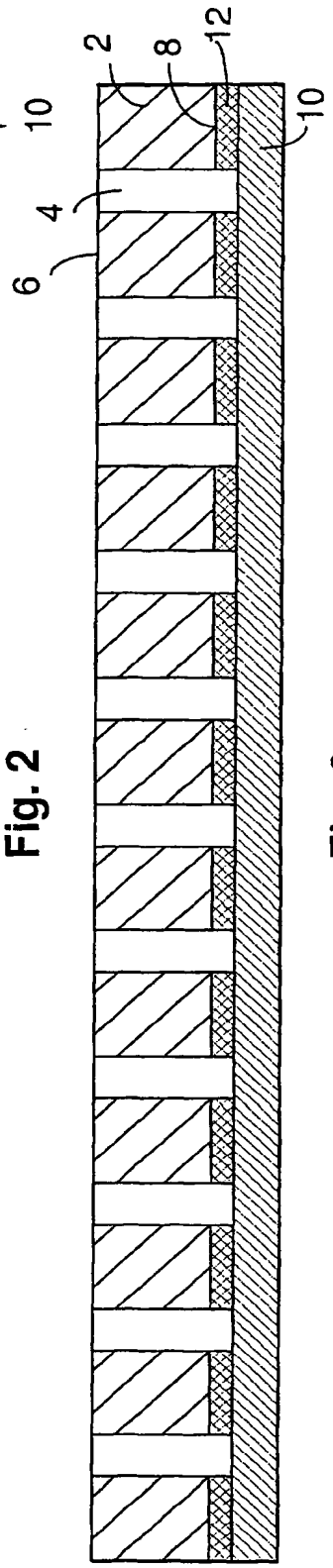


Fig. 3

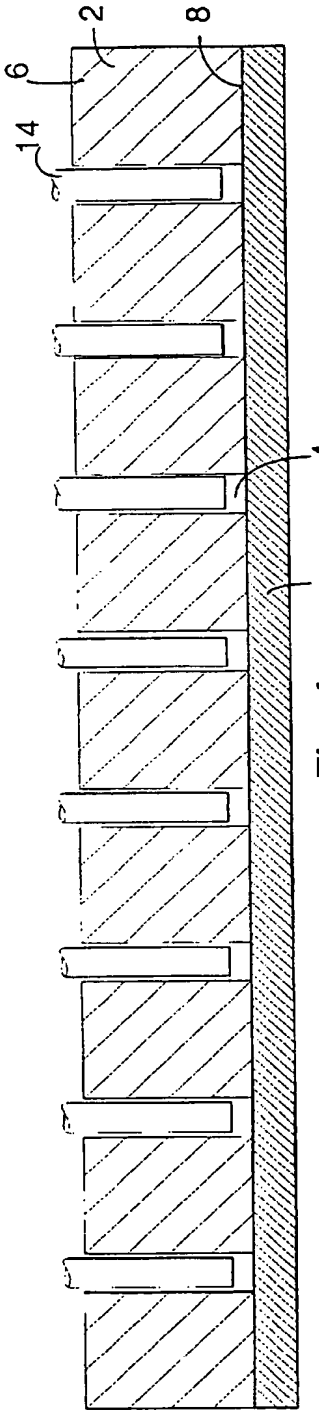


Fig. 4

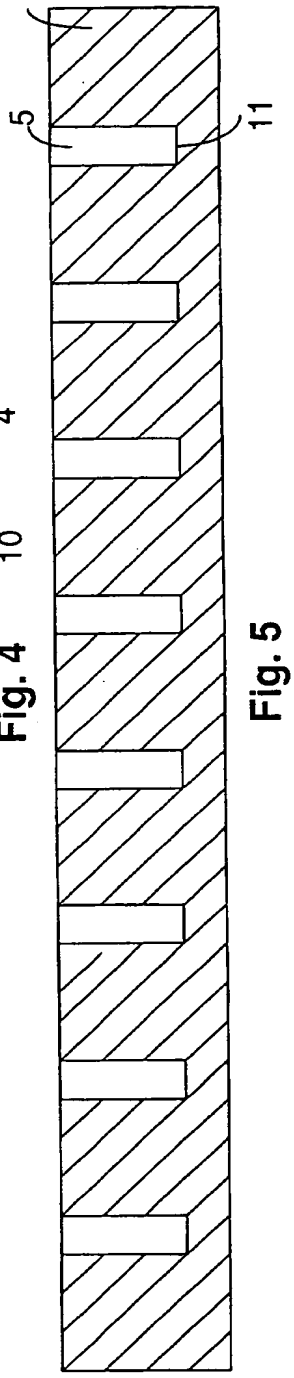


Fig. 5

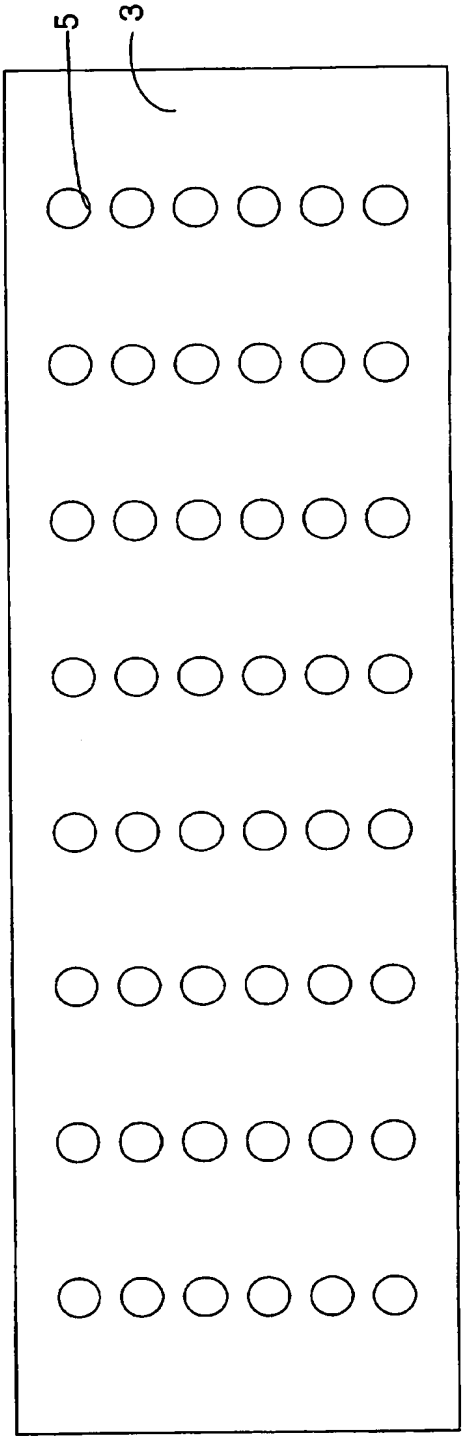


Fig. 6

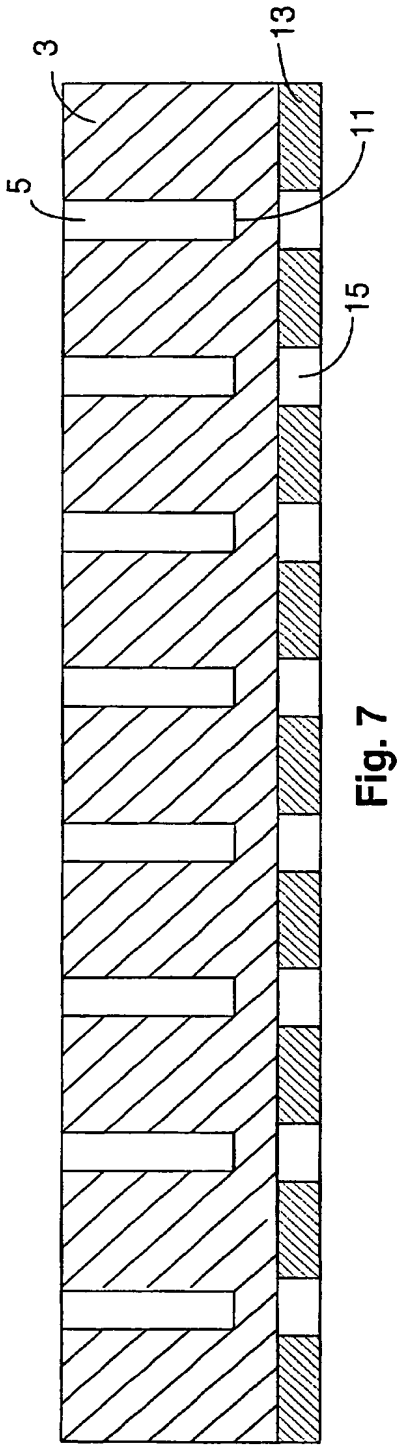


Fig. 7

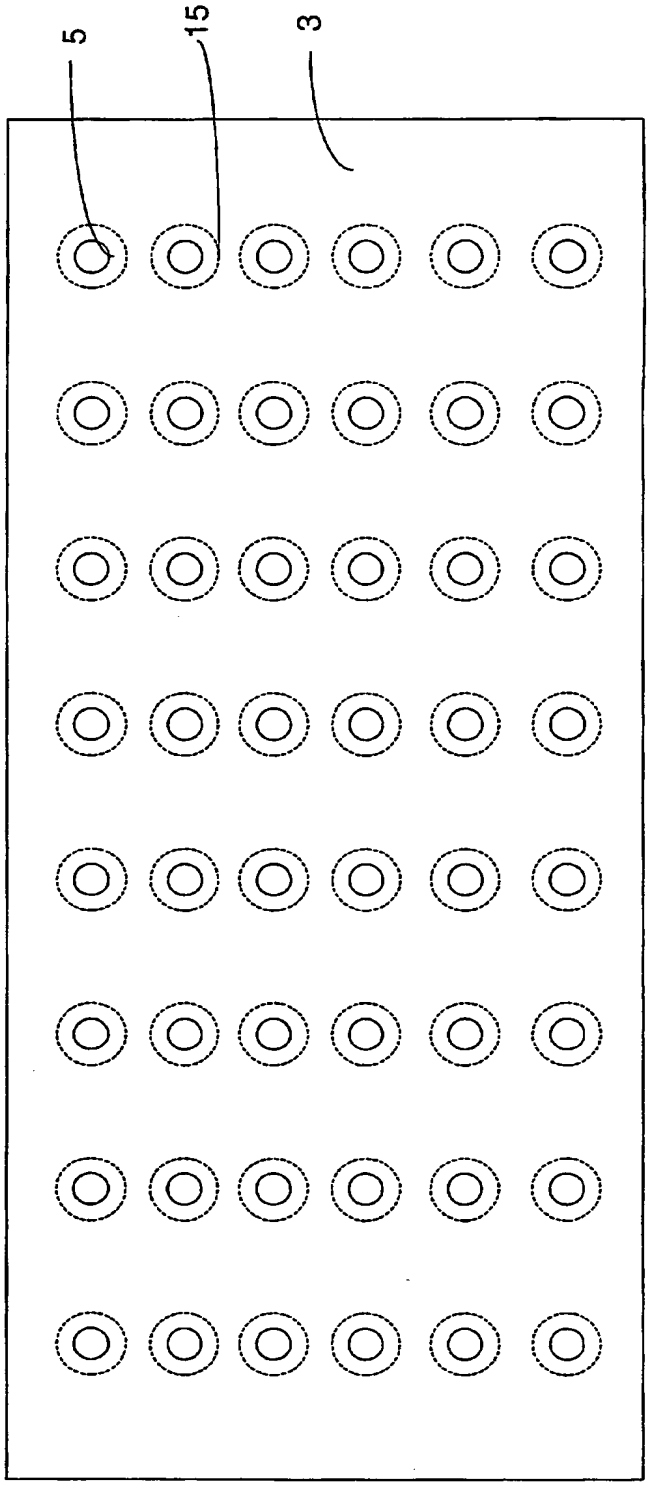


Fig. 8